Effects of Long-Time Exposure in Alloy 625 at

1200°F, 1400°F and 1600°F

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Abstract

An investigation was carried out on alloy 625 to study the structural effects of up to 46,000 hours of aging at 1200°F, 1400°F, and 1600°F using SEM and X-ray diffraction techniques.

Results of this study showed that γ'', delta and αCr phases form at 1200°F in 8,000 hours, and are present after 46,000 hours while delta and M_6C phases form and are present at 1400°F and 1600°F. The formation of γ'' promotes high tensile properties at 1200°F. The decreased amount of delta found at 1600°F indicated that 1600°F is close to the delta solvus temperature in alloy 625. Microstructural results will be presented and correlated to mechanical properties.

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Introduction

Alloy 625 has been among the most successful nickel base superalloys for long time engineering applications. It is typically used for gas turbine engine components, such as combustors, transition ducts, exhaust systems, thrust reverser assemblies and afterburners.[1] Alloy 625 has also demonstrated excellent performance in nuclear power plant operation environments where very reliable long-term performance is required.[2] Its wide use is attributed to its specific combination of fabricability, mechanical properties and corrosion resistance.

In the 70's, a long time aging study was carried out by Matthews in which mechanical properties of alloy 625 were determined for samples exposed 8,000 hours at 1200°F, 1400°F, and 1600°F[3]. It was found that an increase of tensile strength was developed after exposure at 1200°F and then the tensile strength dropped rapidly in samples aged at 1400°F and 1600°F.

The reported X-ray results of this study showed that two M_6C phases and a Ni_3Cb phase were present at 1600°F, but the increased strength at 1200°F was not readily explained by the X-ray data. Because limited microstructural studies of the 1200°F sample were carried out, the increase in strength at 1200°F was not correlated to the microstructure present.

Additional samples of alloy 625 were exposed for 16,000 hours at 1200°F, 1400°F, and 1600°F. Mechanical properties of the 16,000 hour exposed samples showed little change from the mechanical properties developed in 8,000 hours exposure and consequently, very little effort was devoted to understand the structures present.

The aging on the alloy 625 samples was continued for 46,000 hours at 1200°F, 1400°F, and 1600°F. The 46,000 hours samples as well as some samples that were previously exposed for 8,000 hours and 16,000 hours were made available for study of the structural behavior at 1200°F and to note the changes developed in the structures after exposure for 46,000 hours at 1200°F, 1400°F, and 1600°F.

Materials

Plate samples of alloy 625 which were aged 8,000, 16,000 and 48,000 hours at 1200°F, 1400°F, and 1600°F were received from Haynes International. A mill annealed sample as well as a sample aged for 100 hours at 1200°F were included with the aged samples for characterization. The composition of the heat of alloy 625 studied is given in Table 1.

Table 1. Chemical Composition of Alloy 625.

Element	Ni	Fe	Cr	Mo	C	Si	Cb	Al	Ti
Weight %	60.49	3.49	20.67	9.05	.05	.38	3.62	0.25	0.27

Experimental Procedures

All the aged samples were prepared for microstructural evaluation by polishing through 600 grit SiC paper and 6 micron diamond paste. Electropolishing was carried out for 20 seconds at 25 volts in a 20% H_2SO_4–methanol electrolyte. The samples were then electro-etched at 5 volts for 8 seconds in a solution of 170 cc H_2PO_4, 10 cc H_2SO_4 and 15 grams of CrO_3. This electro-etch puts the γ, γ', and delta phases in relief and provides optimum contrast for SEM studies.

Phase extractions were carried out in a 10% HCl-methanol solution at 5 volts for 1 1/2 hours. X-ray diffraction studies to identify the various phases were carried out on the extracted residue.

To monitor the extraction process, SEM studies were carried out on the extracted residue as well as SEM evaluation of the partially extracted residue on the extraction sample. In the case of the samples exposed at 1200°F, it was found that the 10% HCl-methanol solution selectively dissolved a large amount of the small matrix precipitation. In this case the extraction solution was modified and additional extractions were carried out.

Results

Microstructural Analysis

The metallographic preparations used in this study were the same as used for studies of longtime behavior of alloy 718.[4] The CrO_3 electrolytic etch puts γ, γ'' and Ni_3Cb phases in relief but etched out Cr rich phases such as αCr, sigma, and $Cr_{23}C_6$.

The structures present in mill annealed or mill annealed plus 100 hours at 1200°F are shown in Figure 1. There is a semi-continuous grain boundary phase which is probably M_6C. The matrix shows larger MC and M_6C particles. SEM examination at 50,000X failed to show any matrix precipitation due to the 100 hour exposure at 1200°F.

<u>1200°F</u>

When a sample of alloy 625 was given a 10% prestrain and exposed for 8,000 hours at 1200°F, a disk shaped precipitation was found in the grains and a plate-like structure was found predominately at the grain boundaries. Closer examination showed holes to be present at some boundaries which contained heavy precipitation of plates. These representative structures are shown in Figure 2.

Samples of alloy 625 aged for 16,000 hours without prior prestrain showed less plate structures at the grain boundaries and a dense precipitation in the grains, Figure 3. Holes were also present at various grain boundaries.

Aging for 46,000 hours produces more transitions of the matrix precipitates (identified by X-ray analyses to be γ'') to the plate structure (identified as delta Ni_3Cb), Figure 4. The size and number of holes have increased over that found after 16,000 hours.

When the 46,000 hour sample which was used for extraction of phases was evaluated on the SEM, the grain boundary phase appeared to be continuous, delta plates were large and extensive, and the γ'' phase was still heavily precipitated in the grains, Figure 5.

<u>1400°F</u>

The structures formed after 16,000 and 46,000 hours at 1400°F appeared to be delta plates and a continuous grain boundary phase, Figure 6. There were no holes or γ'' matrix precipitation at this temperature after 16,000 and 46,000 hours.

<u>1600°F</u>

The samples aged for 16,000 and 46,000 hours at 1600°F showed vastly different appearing structures than those found at 1200°F and 1400°F. Not only were the γ'' phase and holes absent but the morphology of the delta and grain boundary phase was altered as shown in Figure 7. The plates appeared smaller and somewhat coarsened while the grain boundary phase showed a necklace type of morphology.

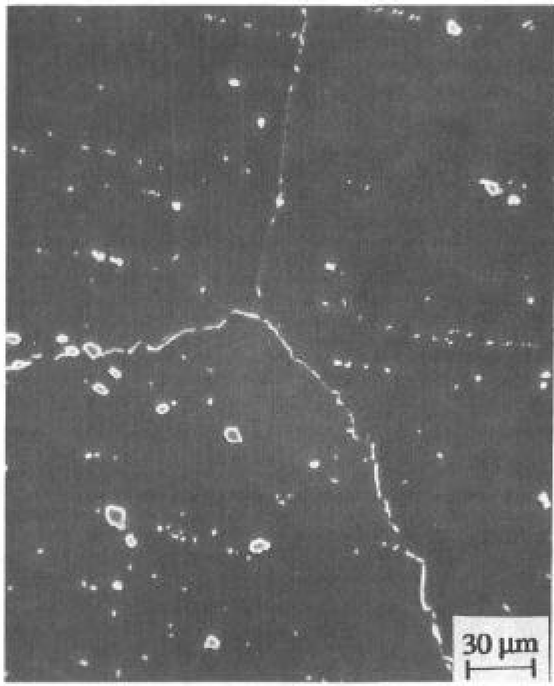
X-Ray Diffraction Results

<u>Mill Annealed</u>

The X-ray pattern for the extracted residue for the mill annealed condition showed a primary MC with a lattice parameter of 4.44Å and an M_6C phase whose parameter in about 10.91Å

<u>1200°F</u>

The X-ray patterns of residues of samples exposed for 8,000 and 16,000 hours show a more complex pattern. In addition to the original M_6C phase, lines in the pattern fit the delta Ni_3Cb phase and an αCr phase. Some of the lines of the Ni_3Cb phase overlap some lines of the M_6C phase to make interpretation more difficult.



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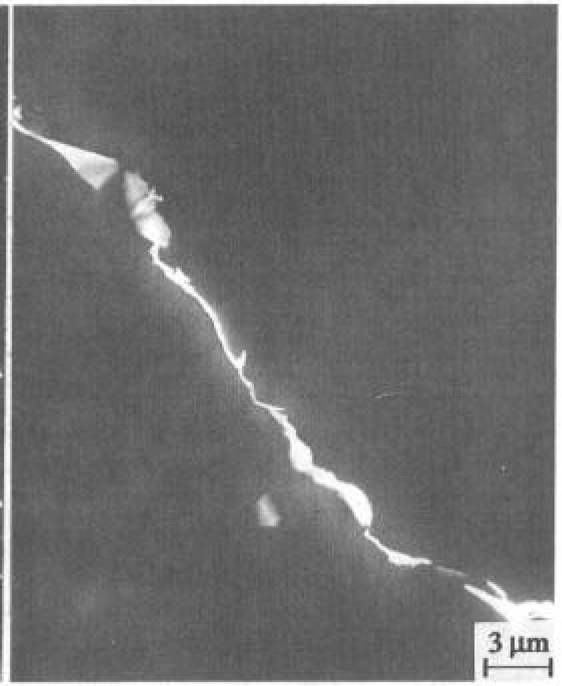

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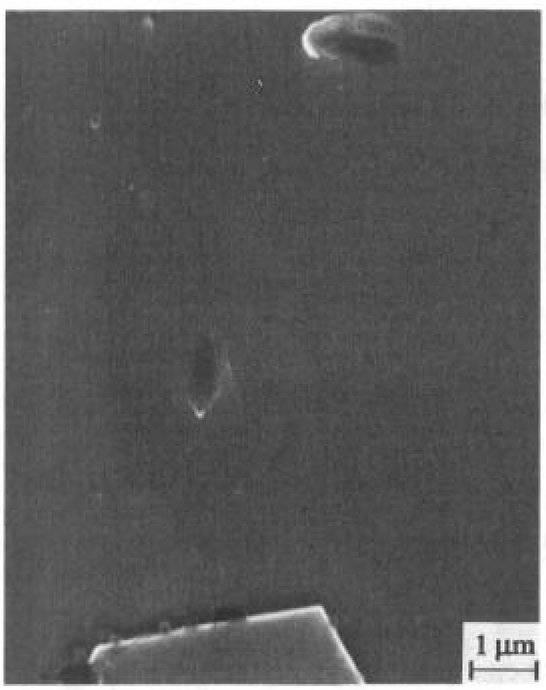

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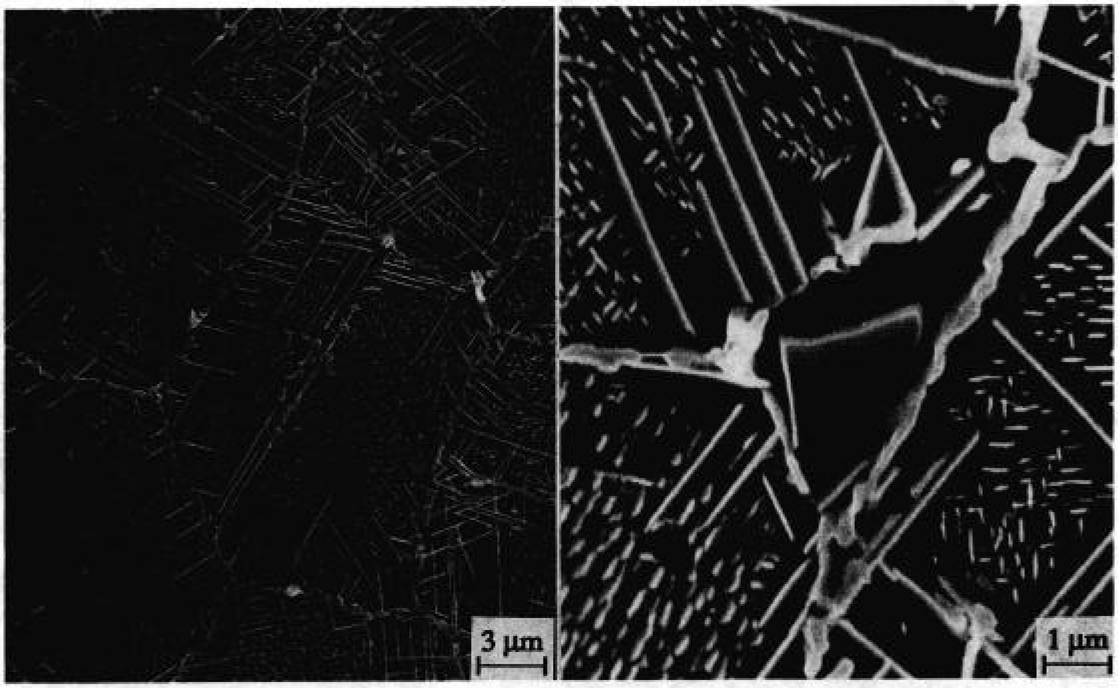
Fig. 1. Mill Anneal + 1200°F/100h.



3K 10K

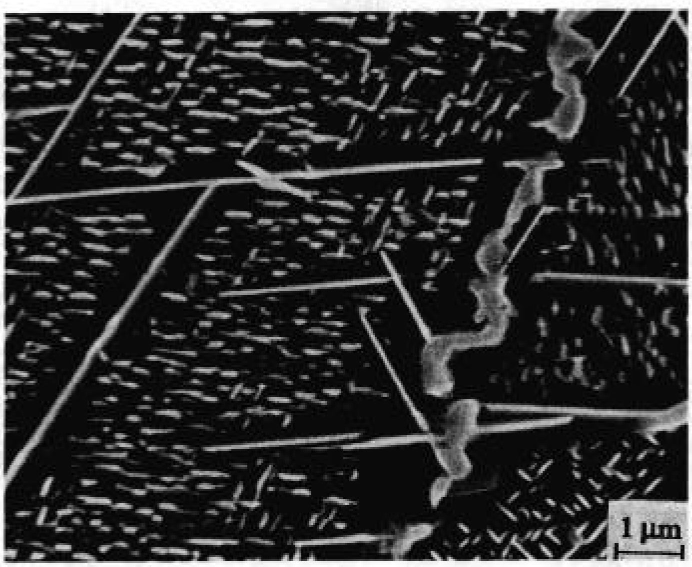


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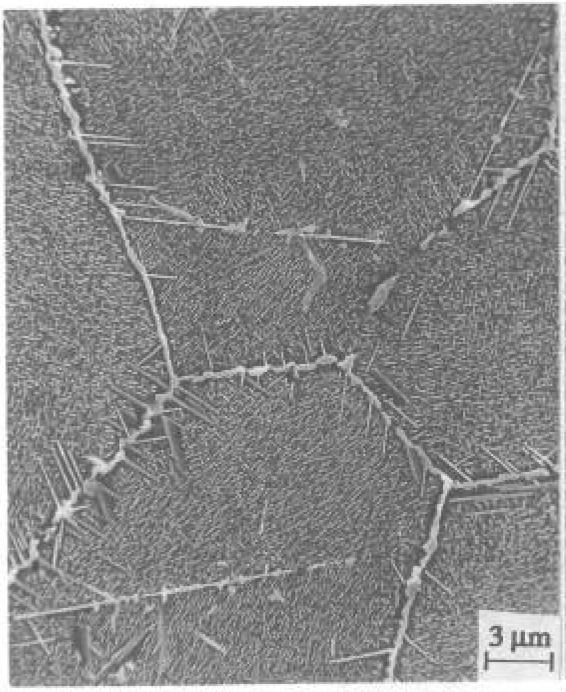
Fig. 2. 1% Prestrain + 1200°F/8000h.



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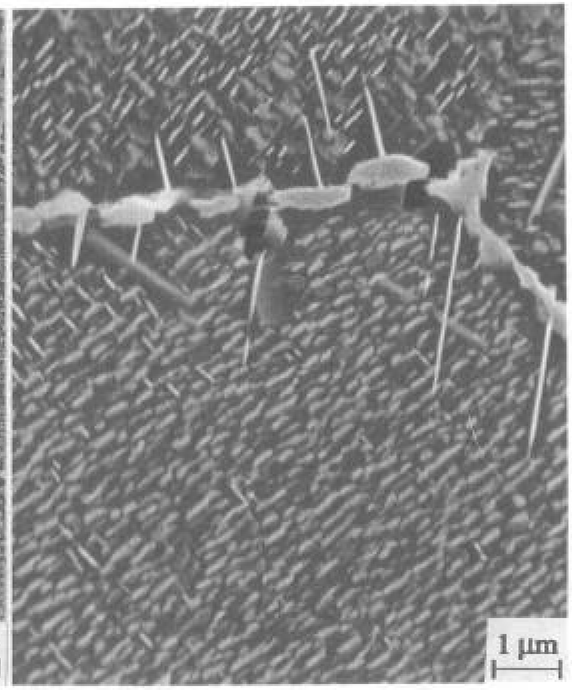

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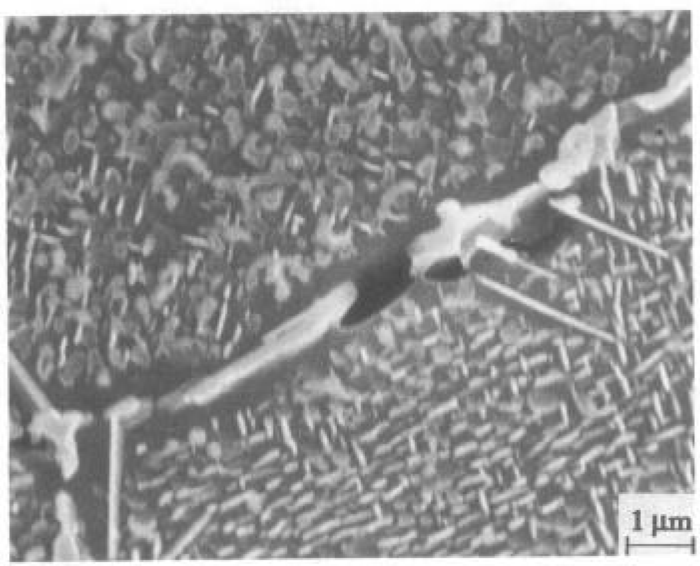

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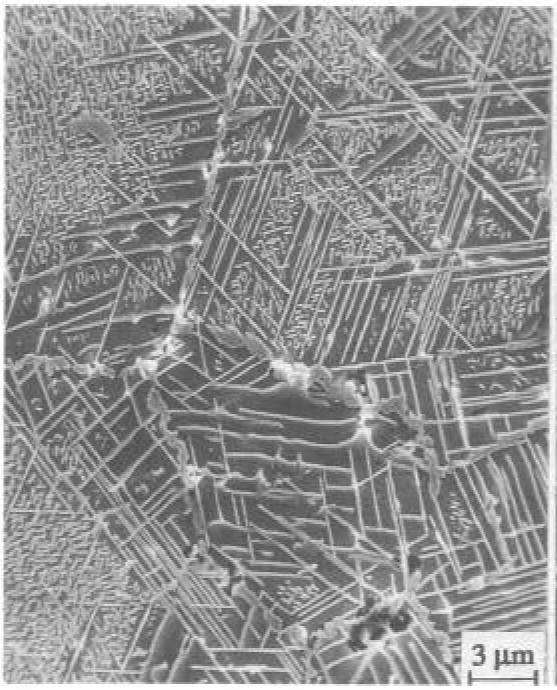
Fig. 3. 0% Prestrain + 1200°F/16000h.



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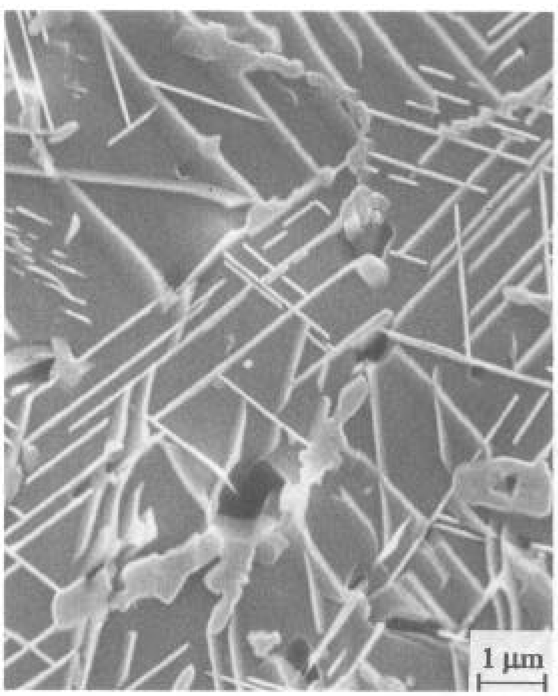

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Fig. 4. 1200°F/46000h.

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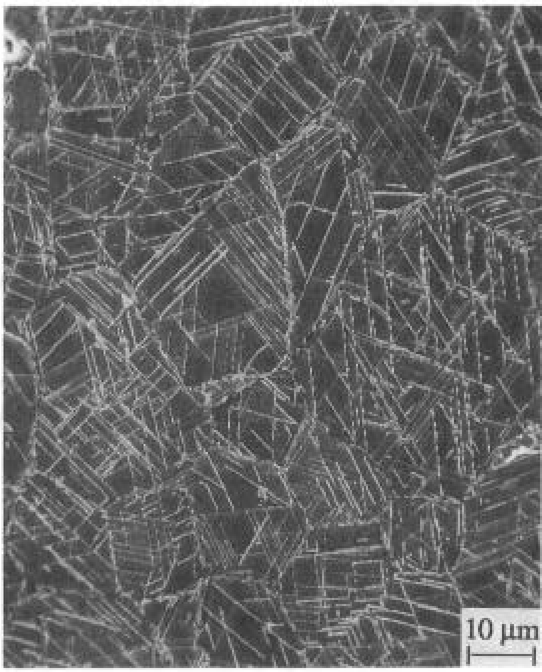
Fig. 5 Extraction Sample
1200°F/46000h



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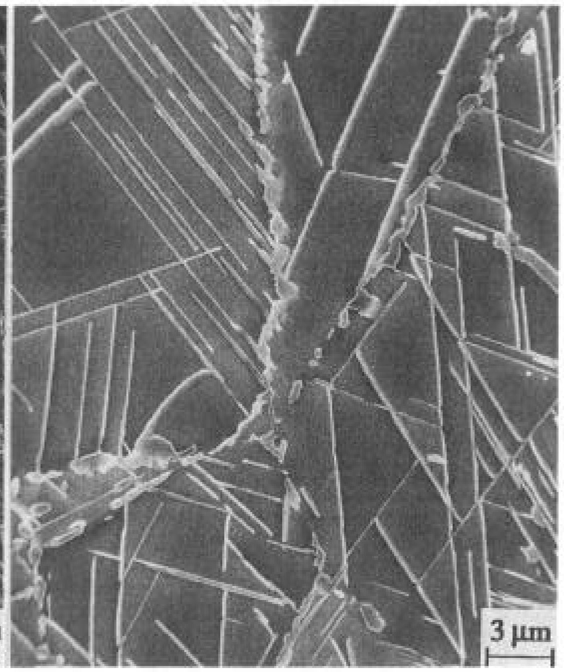

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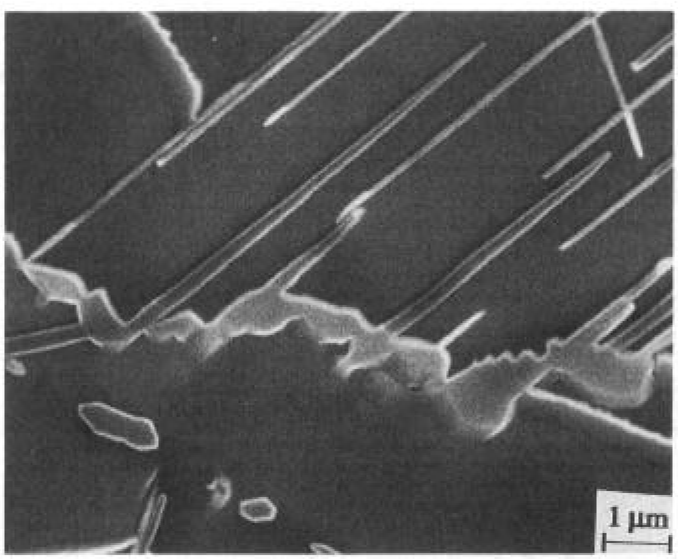

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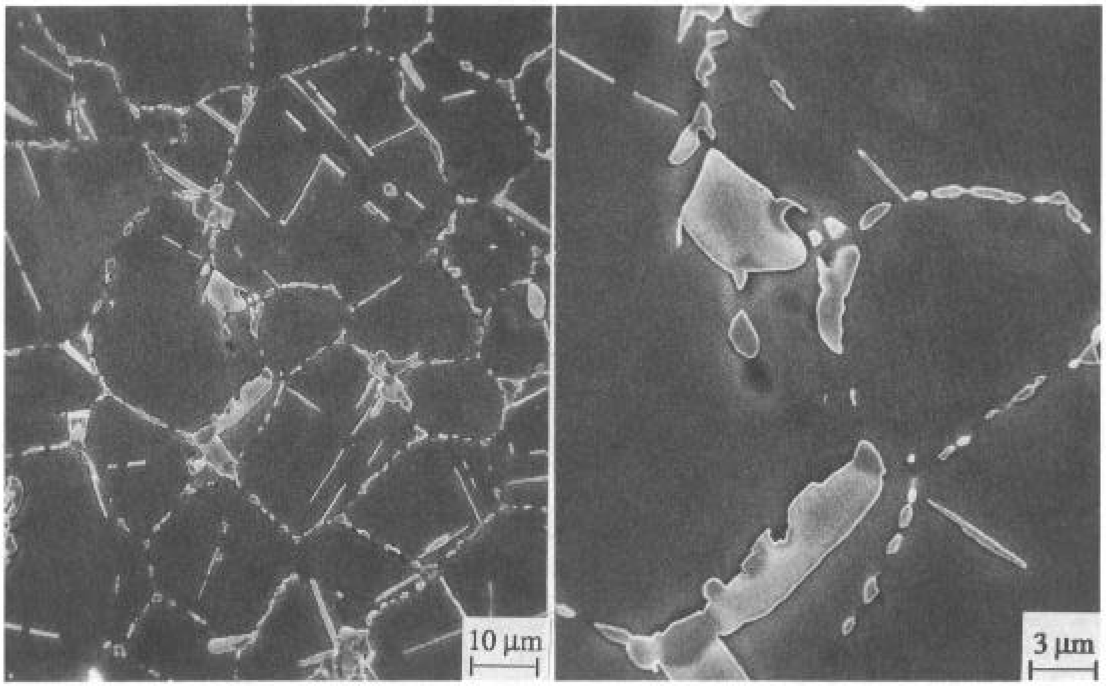
Fig. 6. 1400°F/46000h.



1K 3K

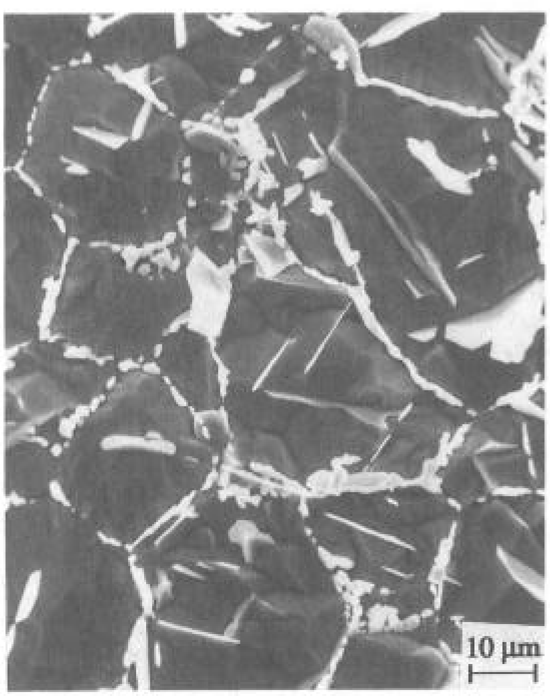


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Fig. 7. 1600°F/46000h.

Studies of the X-ray diffraction results of residues extracted with 10% HCl-methanol did not show the small matrix precipitate seen with the SEM. When the extraction solution was modified, the fine matrix precipitation was found to be γ'.

The extended exposure of 46,000 hours at 1200°F showed that the αCr became much stronger compared to the shorter exposure times. The Ni_3Cb, M_6C, and γ' phases were also identified in this sample.

<u>1400°F</u>

The X-ray study of the phases present after 16,000 and 46,000 hour exposures showed predominantly M_6C and delta Ni_3Cb. A trace of $M_{23}C_6$ could be present but because of all the X-ray lines in the pattern, no positive identification could be made for $M_{23}C_6$.

<u>1600°F</u>

The residues of the 16,000 and 46,000 hour exposures at 1600°F showed only Ni_3Cb and two M_6C phases whose parameters are 10.93 Å and 11.06 Å. Closer examination of the delta phase X-ray pattern showed the delta to be a higher parameter structure than that at 1200°F.

Mechanical Properties

The tensile test properties and the impact data found after 8,000 and 16,000 hours exposure at 1200°F, 1400°F, and 1600°F are courtesy of Haynes International and are presented in Table 2.

The tensile and impact data for the 8,000 and 16,000 hour exposure appear to be similar except for the decrease at 1200°F of the elongation. The decrease of elongation from 18% to 12.1% appears significant and indicates structural changes due to continued exposure at 1200°F.

Table 2. Room Temperature Tensile and Impact Properties

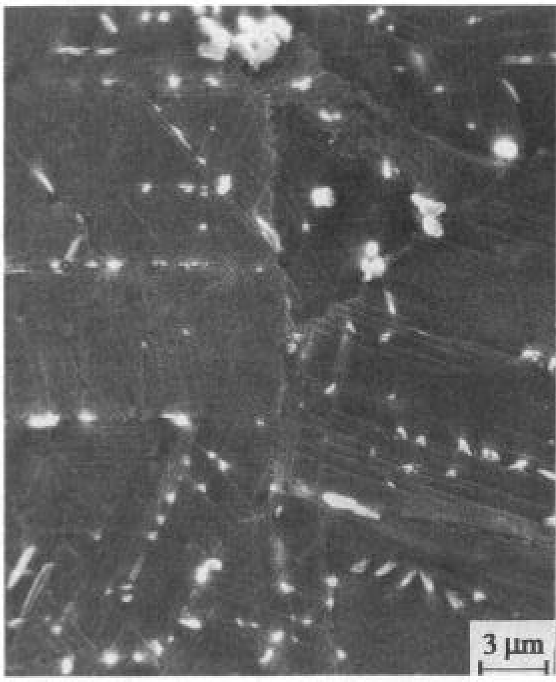
		Y.S.(ksi)	UTS (ksi)	E1%	RA%	Cv(ft.lb)
	Mill Annealed	66.2	127.7	46.1	54.3	81
	1200°F	117.8	164.2	18	17.3	5
8,000 hours	1400°F	97.4	142.6	13.3	13.5	5
	1600°F	63.7	127	26	23	15
	1200°F	118.5	165.4	12.1	12.8	4
16,000 hours	1400°F	96.1	140.4	12.2	11.2	4
	1600°F	63.4	128.4	32.1	27.0	14

Discussion

It appears that the microstructures developed in longtime exposures of alloy 625 at 1200°F are similar to some of those found in alloy 718 in longtime exposures at 1300°F and 1400°F, namely Ni_3Cb, γ', and αCr [4]. Unlike alloy 718, alloy 625 does not form sigma or γ because of the low Fe and Al + Ti contents but forms M_6C due to its high Mo content. In alloy 625 the γ' and αCr phases found at 1200°F are not found at 1400°F and 1600°F. In addition to X-ray identification the absence of αCr at 1400°F and 1600°F was verified by only electropolishing the 46,000 hour exposed samples. The αCr phase is brought into relief and is easily seen in the SEM examination. Figure 8 shows the αCr phase to be present only in the 1200°F exposed sample.

At 1200°F, the γ' transition to delta Ni_3Cb occurs initially at the grain boundary areas. When the material is prestrained prior to aging, the transition occurs more rapidly than if the material were aged for 16,000 hours.

The selective metallographic as well as X-ray analysis techniques used in the study failed to find any sigma phase as might be expected from the high Cr content of alloy 625.



1200°F

1400°F

1600°F

Fig. 8. As Electropolished Only - 46000h.

3K

While delta and M_6C phases were present at 1600°F, the morphologies of these phases at 1600°F are very different than those found at 1200°F and 1400°F. The grain boundary M_6C is discrete and the delta phase in the grains is smaller and lessor in amount.

From similar appearing microstructures found in longtime exposed alloy 718, the presence of considerable γ'' precipitation in alloy 625 found after 46,000 hours aging at 1200°F should provide comparable tensile strength similar to that found after 16,000 hours at 1200°F; however, the development of delta plates at the grain boundaries should decrease the elongation from that found after 16,000 hours.

Conclusions

In alloy 625, longtime exposures of 46,000 hours at 1200°F, 1400°F, and 1600°F produced a variety of different structures. At 1200°F, a γ'' phase was found which transformed to delta Ni_3Cb more rapidly at grain boundaries with exposure time. The transition was accelerated by prior prestrain. An αCr phase was formed at the grain boundaries which continued to increase in amount and size with increased aging time.

At 1400°F, the γ'' and αCr were not found but extensive delta formation occurred in the grains while the grain boundary phase coarsened.

The microstructural pattern changed at 1600°F. Fewer delta plates were found indicating that 1600°F was close to the delta solvus temperature. The massive grain boundary phase which was continuous at 1200°F and 1400°F appeared as a discrete precipitate.

No sigma or γ' phase was found in the alloy 625 as have been found in longtime aged alloy 718. Based on similar microstructures and their effects on tensile and impact properties, the 1200°F aged material should still have high tensile properties but should see a decrease of elongation due to delta formation at the grain boundaries.

Acknowledgements

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References

1. S. J. Matthews, "Thermal Stability of Solid Solutioned Strengthened High Performance Alloys," 3rd International Superalloy Symposium, Seven Springs, PA (1976).
2. Copson, H. R. and Economy, G. (1968). Effect of Some Environmental Conditions on Stress Corrosion Behavior of Ni-Cr-Fe Alloys in Pressurized Water. Corrosion 24 (3), No. 3: 55.
3. S. J. Matthews, "Thermal Stability of Solid Solutioned Strengthened High Performance Alloys (16000 hour data)," (Haynes Internal Report No. 9265, (1977)).
4. J. F. Radavich and G. E. Korth, "High Temperature Degradation of Alloy 718," Superalloys 1992, pp. 497-506.